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REEL #

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RYUSHCHEN, V. YE.

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S/063/60/005/003/005/011/XX  
A051/A029

5.2200 1043, 1273, 1228

AUTHORS: Yuranova, L.I., Komissarova, L.N., Plyushchev, V.Ye.

TITLE: On the Formation of Hexahydrates of Zirconium and Hafnium Oxynitrates

PERIODICAL: Zhurnal Vsesoyuznogo Khimicheskogo Obshchestva im. D.I.  
Mendeleeva, 1960, Vol. 5, No. 3, p. 346

TEXT: The authors recently conducted a study on a new method for synthesizing  $ZrO(NO_3)_2 \cdot 2H_2O$  and a similar compound of Hafnium, i.e., the dihydrates of zirconium and hafnium oxynitrates. It is possible to obtain individual zirconium and hafnium oxynitrates free of admixtures of any other nitrates. The results could be successfully reproduced several times. Numerous experiments showed that both substances crystallize in a wide range of  $HNO_3$  concentrations and of the zirconium and hafnium concentration, as well as under various temperature conditions. While studying the system

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$ZrO_2-N_2O_5-H_2O$  at  $25^\circ C$  by the physico-chemical method of analysis, the existence of another compound was established, viz., the hexahydrate of zirconium oxynitrate with the composition:  $ZrO(NO_3)_2 \cdot 6H_2O$ . The compound crystallizes from solutions containing from 21.35 weight %  $ZrO_2$  and 1.93 weight % of  $N_2O_5$  to 4.50 weight % of  $ZrO_2$  and 40.00 weight % of  $N_2O_5$ . The hexahydrate of hafnium oxynitrate can be formed in the same way. The formation of the latter two compounds also takes place when a small excess of  $ZrO(NO_3)_2 \cdot 2H_2O$  and  $HfO(NO_3)_2 \cdot 2H_2O$  is introduced into a saturated solution of these compounds, or by mixing the dihydrate of zirconium and hafnium oxynitrates with a given amount of water, which is insufficient for the complete dissolution of the initial salts (the molecular ratio of the water and the dihydrates changed from 2:1 to 8:1). In the first case, the crystallization of the hexahydrates, carried out in glass containers for studying the solubility, continued for 8-10 hours in the entire volume of the solution, so that a completely solidified mass was obtained, which was

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difficult to crush and which resembled magnesium cement. In the second case mixtures were obtained in the form of a gruel, which after a certain time hardened into a non-transparent, white, enamel-like mass. The obtained preparations were thoroughly ground, dried in air to a constant weight and were analyzed for  $\text{MeO}_2$ ,  $\text{N}_2\text{O}_5$  and  $\text{H}_2\text{O}$  content. The  $\text{MeO}_2$  content was determined by calcinating the corresponding hydrates to  $\text{MeO}_2$  at  $900^\circ\text{C}$ . The Devard method was used for determining  $\text{N}_2\text{O}_5$ . The amount of water was calculated from the difference. The results of the analysis are given in the table and represent the average values of 5 determinations. It is pointed out that in repeated experiments the results were systematically and favorably reproduced. Thus, the composition of the obtained compounds is expressed by:  $\text{ZrO}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$  and  $\text{HfO}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ . X-ray findings were also obtained, which confirmed the formation of new phases. The hexahydrates were found to be well soluble in water and stable in air. Their densities at  $20^\circ\text{C}$  were estimated pycnographically and found to be  $2.08 \pm 0.02$  and  $2.66 \pm 0.02$ , respectively. There is 1 table and 3 non-Soviet references.

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On the Formation of Hexahydrates of Zirconium and Hafnium Oxynitrates

ASSOCIATION: Moskovskiy institut tonkoy khimicheskoy tekhnologii im.  
M.V. Lomonosova (Moscow Institute of Fine Technology, im.  
M.V. Lomonosov)

SUBMITTED: December 28, 1959

Table:

Preparation	Content, weight %			Molar ratio $\text{MeO}_2:\text{N}_2\text{O}_5:\text{H}_2\text{O}$
	$\text{MeO}_2$	$\text{N}_2\text{O}_5$	$\text{H}_2\text{O}$	
zirconium nitrate	36.30	31.85	31.85	1.0:1.02:6.1
hafnium nitrate	50.00	25.11	24.89	1.0:0.98:5.9

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S/063/60/005/003/002/003

A003/A001

5.2100

AUTHORS: Grizik, A.A., Plyushchev, V.Ye.

TITLE: On the Synthesis of Lithium Metazirconate and Metahafnate

PERIODICAL: Zhurnal vsesoyuznogo khimicheskogo obshchestva im.D.I. Mendeleeva, 1960, Vol. 5, No. 3, pp. 349 - 350

TEXT: There are literature data (Refs 2-8) on the existence of three lithium zirconates, but the existence of only one, viz.,  $\text{Li}_2\text{O} \cdot \text{ZrO}_2$  was proved. This zirconate is used in the silicate industry (Ref 9). High-temperature synthesis was used here to obtain pure zirconates, and their properties were studied.  $\text{ZrO}_2$  (with a content of 0.03%  $\text{HfO}_2$ ) and  $\text{HfO}_2$  (with a content of  $< 1\%$   $\text{ZrO}_2$ ) were employed as initial materials. Chemically pure  $\text{Li}_2\text{CO}_3$  was used instead of  $\text{Li}_2\text{O}$ . It decomposes at high temperatures to  $\text{Li}_2\text{O}$ . The experiments were carried out at 800-1,200°C. Above 900°C alundum crucibles were used, because platinum vessels are destroyed. The analysis of the reaction products showed that the composition of the products obtained at a  $\text{Li}_2\text{O}:\text{MeO}_2$  ratio from 1:1 to 1:4 corresponds to the formula  $\text{Li}_2\text{MeO}_3$ . Pure meta-compounds are obtained in the interaction of  $\text{MeO}_2$  and  $\text{Li}_2\text{CO}_3$  (taken in the ratio  $\text{Li}_2\text{O}:\text{MeO}_2 = 1:1$ ) at  $1,100 \pm 20^\circ\text{C}$ . The analysis by the

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weight method showed the following results (weight %) found:  $\text{Li}_2\text{O}$  19.54;  $\text{ZrO}_2$  80.82.  $\text{Li}_2\text{ZrO}_3$ ; calculated  $\text{Li}_2\text{O}$  19.52;  $\text{ZrO}_2$  80.48. Found  $\text{Li}_2\text{O}$  12.50;  $\text{HfO}_2$  87.52.  $\text{Li}_2\text{HfO}_3$ ; calculated  $\text{Li}_2\text{O}$  12.43;  $\text{HfO}_2$  87.57. Crystalline powders of white color with a high refractive index are obtained. The following densities were determined by the pycnometric method (20°C):  $\text{Li}_2\text{ZrO}_3$   $4.123 \pm 0.003$ ;  $\text{Li}_2\text{HfO}_3$   $6.453 \pm 0.003$ . Both compounds melt above 1,500°C and are not hygroscopic.  $\text{Li}_2\text{MeO}_3$  is hydrolyzed by water. Metazirconate and metahafnate are decomposed by boiling in diluted (1:1)  $\text{HCl}$  and  $\text{H}_2\text{SO}_4$ , concentrated  $\text{H}_2\text{SO}_4$  decomposes them in the cold. X-rays cause an intensive light-blue luminescence of  $\text{Li}_2\text{HfO}_3$ . There are 11 references: 2 Soviet, 3 English, 3 German, 2 American and 1 Canadian. X

ASSOCIATION: Moskovskiy institut tonkoy khimicheskoy tekhnologii im. M.V. Lomonosova  
(Moscow Institute of Fine Chemical Technology imeni M.V. Lomonosov)

SUBMITTED: November 14, 1959

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69051

5.2340  
5.2300

AUTHORS:

Komissarova, L. N., Plyushchev, V. Ye., S/078/60/005/03/014/048  
Kremenskaya, I. N. B004/B002

TITLE:

Investigation as to Solubility and Thermal Stability of Zirconium Oxychloride

PERIODICAL:

Zhurnal neorganicheskoy khimii, 1960, Vol 5, Nr 3, pp 586-592 (USSR)

ABSTRACT:

First, the authors give a survey of publications on  $ZrOCl_2 \cdot 8H_2O$ , and mention L. K. Akhrap-Simonova (Ref 15). According to it, there are discrepancies as to solubility and stability of this compound, which urged the authors to write the present paper. The investigation was carried out with spectrally pure  $ZrOCl_2 \cdot 8H_2O$ . The removal of  $HfO_2$  was achieved by means of ion exchange chromatography. The solubility was investigated in the Wobser ultrathermostat type U-8 (Table 1, Fig 1) within the range  $-2$  to  $+110^\circ$ . It was found that the solubility of  $ZrOCl_2 \cdot 8H_2O$  increases with the temperature being raised to  $70.5^\circ$ . The maximum is reached at 42.00 weight% of  $ZrO_2$ . Below  $70.5^\circ$  no hydrolysis was observed in the concentrated solutions, as was observed by I. V. Tananayev and L. S. Guzeyeva (Ref 21) in diluted solutions. Above  $70.5^\circ$  the solubility of zirconium oxychloride is reduced due to hydrolysis and the development of compounds with a low content of chlorine of the general composition  $mZrOCl_2 \cdot nZrO(OH)Cl \cdot pH_2O$  ( $m > n$ ,  $p < 8$ ). At  $110^\circ$ , a homogeneous, viscous

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Investigation as to Solubility and Thermal Stability of Zirconium Oxychloride

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polymerization product develops as hitherto had only been observed in zirconium nitrates. The thermal stability of  $ZrOCl_2 \cdot 8H_2O$  was investigated by means of the following methods: 1) heating to a certain temperature under the influence of air until constancy of weight is attained, 2) taking of thermograms by means of an N. S. Kurnakov pyrometer, and 3) by means of continuous weighing. The results are shown by tables 2, 3, and figures 2, 3. A partial loss of water already sets in at  $45^\circ - 65^\circ$ . At  $80^\circ$  decomposition sets in accompanied by the loss of chlorine, taking place in three stages and being finished at  $400^\circ - 450^\circ$ . Table 4 compares the results obtained according to various methods. Slight differences in the data are caused by different rates of heating. There are 3 figures, 4 tables, and 21 references, 3 of which are Soviet.

ASSOCIATION:

Moskovskiy institut tonkoy khimicheskoy tekhnologii im. M. V. Lomonosova (Moscow Institute of Fine Chemical Technology imeni M. V. Lomonosov). Khimicheskii fakul'tet MGU (Chemical Department of the Moscow State University)

SUBMITTED:

November 3, 1958

Card 2/2

AUTHORS: Tulinova, V. B., Plyushchev, V. Ye., S/078/60/005/03/033/048  
Ternovskaya, I. V., Lukova, S. N. B004/B005  
Samuseva, R. G.

TITLE: Investigation of the Joint Solubility of Lanthanum and Sodium Sulfates

PERIODICAL: Zhurnal neorganicheskoy khimii, 1960, Vol 5, Nr 3, pp 695-700 (USSR)

ABSTRACT: The present paper is part of an extensive investigation of the physicochemical foundation of methods of separating rare earths which was started together with G. G. Urazov (Ref 3). The solubility in the system  $\text{La}_2(\text{SO}_4)_3 - \text{Na}_2\text{SO}_4 - \text{H}_2\text{O}$  was determined at 25, 50, and 75°. The binary systems which are components of this system have been described in publications. The solubility was determined by the isothermal method. The equilibrium between solution and precipitate was established after 14 days which was checked analytically. The sulfate ion was determined gravimetrically as  $\text{BaSO}_4$ , the lanthanum ion either gravimetrically as oxalate or, at low concentrations, colorimetrically according to reference 10. The results are shown in tables 1-3 (for 25, 50, and 75°), and as a diagram in figure 1. One double salt  $\text{La}_2(\text{SO}_4)_3 \cdot \text{Na}_2\text{SO}_4 \cdot 2\text{H}_2\text{O}$  forms

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in the system investigated; its thermogram is shown in figure 2,  
its Debye-pattern data in table 4. There are 1 figure, 4 tables,  
and 11 references, 6 of which are Soviet. (C)

ASSOCIATION: Moskovskiy institut tonkoy khimicheskoy tekhnologii im. M. V.  
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SUBMITTED: November 5, 1958

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PLYUSHCHEV, V.Ye.; SHKLOVER, L.P.; ROZDIN, I.A.

Synthesis of the dithiocyanates of zirconium and hafnium. Zhur.neorg.  
khim. 9 no.1:125-127 Ja '64.  
(MIRA 17:2)

SHAKHNO, I.V.; PLYUSHEV, V.Ye.

Fusibility diagrams in systems formed by rubidium and cesium chlorides with calcium chloride. Zhur.neorg.khim. 5 no.5: 1172-1173 My '60. (MIRA 13:7)

1. Moskovskiy institut tonkoy khimicheskoy tekhnologii im. M.V.Lomonosova.

(Rubidium chloride) (Cesium chloride)  
(Calcium chloride)

S/078/60/005/007/042/043/XX  
B004/B060

AUTHORS: Stepin, B. D., Tartakovskaya, A. M., Plyushchev, V. Ye.

TITLE: Reversibility of the Lyotropic Series of Alkali Metals

PERIODICAL: Zhurnal neorganicheskoy khimii, 1968, Vol. 5, No. 7.  
pp. 1612-1617

TEXT: The authors attempted to ascertain whether the reversibility of the lyotropic series  $Cs^+ > Rb^+ > K^+ > Na^+ > Li^+$  on phosphoric acid cation exchangers, claimed in publications (Refs. 4-6), was really possible. This phenomenon would be important for the industrial cleaning of rubidium salts from potassium impurities. The authors carried out their tests with a phosphoric acid cation exchanger of the type  $P\ddot{t}$  (RF), which was pre-treated in compliance with ГОСТ 5695-53 (GOST 5695-53). Mixtures consisting of equal volumes of KCl and RbCl solutions were introduced into a column filled with RF in H form, and after 24 hours the column was washed out by means of 0.1 N HCl at a rate of 0.4 ml/min. K and Rb were determined in the eluate by a flame photometer consisting of atomizer, UM-2 (UM-2) monochromator, БЭИ (VEI) photomultiplier, and mirror galvanometer. A

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reversal of the lyotropic series was not observed. Potassium was eluted earlier than rubidium. Separation is rendered difficult due to the small distance between the two fronts. At a ratio of  $KCl : RbCl = 1 : 9$ , a drop in the sorption isotherm was only observed at the rear front of K. Similarly, no reversal was established in methanol solution or at increased temperature. The authors found in the course of their experiments that on conversion of the cation exchanger into Rb form not all hydrogen ions are replaced by rubidium, although there was the same rubidium concentration both at the inlet and outlet of the column. They explain this by ion exchange between the functional groups of surface and interior of exchanger grains. The RF cation exchanger contained acid groups with different degrees of dissociation. There are 6 figures, 2 tables, and 9 references: 5 Soviet and 4 US.

ASSOCIATION: Moskovskiy institut tonkoy khimicheskoy tekhnologii im. M. V. Lomonosova, Kafedra tekhnologii redkikh i rasseyannykh elementov (Moscow Institute of Fine Chemical Technology imeni M. V. Lomonosov, Chair of Technology of Rare and Trace Elements)

SUBMITTED: March 27, 1959

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TULINOVA, V.B.; STATSENKO, A.A.; PLYUSHCHEV, V.Ye.

Joint solubility of potassium hydroxide and potassium sulfate.  
Zhur. neorg. khim. 5 no.11:2530-2533 N '60. (MIRA 13:11)

1. Moskovskiy institut tonkoy khimicheskoy tekhnologii imeni M.V.  
Lomonosova.

(Potassium hydroxide)

(Potassium sulfate)



STEPIN, B.D.; PLYUSHCHEV, V.Ye.

Determination of small amounts of potassium in rubidium chloride by means of flame photometry. Zhur. anal. khim. 15 no.5:556-560 3-0 '60. (MIRA 13:10)

1. M.V. Lomonosov Moscow Institute of Fine Chemical Technology.  
(Potassium--Analysis) (Rubidium chloride)  
(Flame photometry)

5.2100

77492  
SOV/80-33-1-1/49

AUTHOR: Plyushchev, V. Ye.

TITLE: Concerning the Theory of Sulfate Method for Processing of Spodumene

PERIODICAL: Zhurnal prikladnoy khimii, 1960, Vol 33, Nr 1, pp 3-9 (USSR)

ABSTRACT: The author studied the reaction between spodumene and  $\text{Na}_2\text{SO}_4$  in the temperature range of 700-1,100° in order to establish the effect of temperature and quantity of  $\text{Na}_2\text{SO}_4$  upon the yield of lithium, and to investigate the insoluble reaction products. The reaction mixture consisted of 5-10 g of  $\alpha$ - or  $\beta$ -spodumene (the  $\alpha$ -form,  $(\text{Li}, \text{Na})\text{AlSi}_2\text{O}_6$ , contained 6.05%  $\text{Li}_2\text{O}$  and 0.12%  $\text{Na}_2\text{O}$ ; the  $\beta$ -form,  $(\text{Li}, \text{Na})\text{AlSi}_2\text{O}_6$ , contained 5.44%  $\text{Li}_2\text{O}$  and 3.20%  $\text{Na}_2\text{O}$ ) mixed with various quantities (20, 40, 60, 80, 100 weight % to the weight of the mineral)

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of  $\text{Na}_2\text{SO}_4$ . The samples, placed into corundum crucibles, were subjected to 3 hours sintering in a muffle furnace. The soluble  $\text{Li}_2\text{SO}_4$  was washed out of the crushed sinters with water. The solutions were analyzed for Li and  $\text{SO}_4^{2-}$ . Figure 1 illustrates effect of temperature and quantity of  $\text{Na}_2\text{SO}_4$  upon the yield of lithium from  $\beta$ -spodumene. It can be seen that intensive reaction begins only at  $900^\circ$ ; i.e., above the melting temperature. While at temperatures below  $900^\circ$  the yield of Li is higher than in sintering with  $\text{K}_2\text{SO}_4$  /Plyushchev, V. Ye., Doklady Akad. nauk SSSR 124.642 (1959); Zhur. Priklad. Khim., 32, 7, 1413 (1959). --Na-ions are used up only in isomorphous exchange with lithium--, at higher temperatures it is substantially lower than for  $\text{K}_2\text{SO}_4$ , due to the low melting point of  $\text{Na}_2\text{SO}_4$ , which leads to liquation of

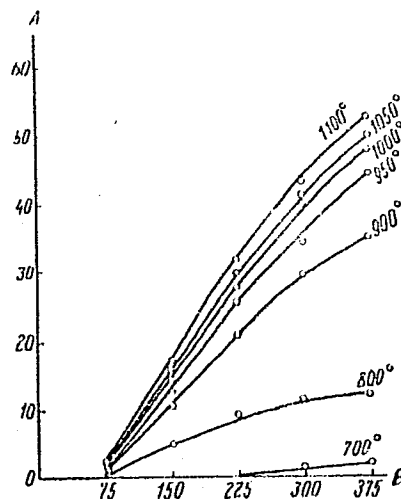
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Fig. 1. Dependence of lithium recovery from  $\beta$ -spodumene upon the temperature of its interaction with  $\text{Na}_2\text{SO}_4$  and expenditure of the reagent: (A) amount of dissolved  $\text{Li}_2\text{O}$  (in %); (B) expenditure of  $\text{Na}_2\text{SO}_4$  (in % of the theoretical).



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the sulfate and to formation of a viscous mass. The cooled melt consists of a brittle salt mass ( $\text{Li}_2\text{SO}_4$ ,  $\text{Na}_2\text{SO}_4$ ) and insoluble residue--rigid, glass-like product which retains almost half of the lithium. X-ray study of the insoluble melt residues at  $1,100^\circ$  have disclosed the presence of  $\text{Al}_2\text{O}_3$ , and thus, decomposition of spodumene at that temperature (chemical analysis of solutions from sinters obtained above  $1,000^\circ$  showed presence of Al and Si). Formation of  $\text{Li}_2\text{SO}_4$  upon sintering of  $\alpha$ -spodumene began only above  $900-950^\circ$ . After  $950^\circ$  the results were very similar to those with  $\beta$ -spodumene. Apparently, heating  $\alpha$ -spodumene leads to its conversion into the  $\beta$ -modification. There are 2 figures; 1 table; and 20 references, 8 Soviet, 1 Swedish, 1 French, 4 German, 2 U.K., 4 U.S. The 5 most recent U.K. and U.S. references are: R. Hader, R. Nielsen,

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M. Herre, Ind. Eng. Ch., 43, 2636 (1951); R. B.  
Ellestad, K. M. Leute, Canad. Pat. 504477 (1954); H. H.  
Hutte, Brit. Pat. 40936 (1933); K. A. Sivander, S. J.  
Wallden, Am. Pat. 2230167 (1941), Ch. A., 35,3043 (1941);  
N. L. Bewen, Am. J. Sci., 33, 1 (1937).

ASSOCIATION:

Moscow M. V. Lomonosov Institute of Fine Chemicals  
Technology (Moskovskiy institut tonkoy khimicheskoy  
tekhnologii imeni M. V. Lomonosova)

SUBMITTED:

May 11, 1959

Card 5/5

KOMISSAROVA, L.N., kand. khim. nauk, red.; PLYUSHCHEV, V.Ye., kand.  
khim. nauk, red.; L'VOVA, I., red.; KHAR'KOVSKAYA, L., tekhn.  
red.

[Methods for the separation of rare earth metals; collection of  
translated articles] Metody razdeleniia redkozemel'nykh metallov;  
sbornik statei. Moskva, IZd-vo inostr. lit-ry, 1961. 361 p.  
(MIRA 15:2)

(Rare earth metals)

S/153/61/004/001/005/009  
B110/B203

AUTHOR: Plyushchev, V.Ye.

TITLE: Study of the reaction of minerals containing rare alkali elements with salts and oxides in sintering and melting processes

PERIODICAL: Izvestiya vysshikh uchebnykh zavedeniy. Khimiya i khimicheskaya tekhnologiya, v. 4, no. 1, 1961, 116 - 121

TEXT: The physicochemical processes in the lithium salt production from spodumene by sintering with  $K_2SO_4$  are still insufficiently known. The great technological importance of this procedure, however, asks for a thorough knowledge of its physicochemical fundamentals. As in the conventional lithium production methods, the mineral was first thermally enriched (decrepitation), and the natural  $\alpha$ -spodumene transformed to  $\beta$ -spodumene by annealing at  $1100^\circ C$ . Mg, Mn, Fe, small amounts of Be, Cu, Zr, Ti, Sn, and Ag traces were also detected by spectrum analysis. A comparison of its analysis with the theoretical, pure composition  $Li[AlSi_2O_6]$  showed the usual,

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partial, isomorphous substitution of Li by Na according to the formula:  $(\text{Li,Na})_2\text{O} \cdot \text{Al}_2\text{O}_3 \cdot 4\text{SiO}_2$ , structural formula  $(\text{Na})[\text{AlSi}_2\text{O}_6]$ . The pure, homogeneous ( $n' = 1.520 \pm 0.003$ ) mineral, free from foreign minerals, passed with 90% a 150-mesh screen. The exchange reaction  $\text{Li}_2\text{O} \cdot \text{Al}_2\text{O}_3 \cdot 4\text{SiO}_2 + \text{K}_2\text{SO}_4 = \text{Li}_2\text{SO}_4 + \text{K}_2\text{O} \cdot \text{Al}_2\text{O}_3 \cdot 4\text{SiO}_2$  does not reflect the qualitatively different structural formations in sintering. The individual phases in mechanical  $\beta$ -spodumene -  $\text{K}_2\text{SO}_4$  mixtures, sintered material obtained from them, and residues left behind after aqueous extraction, were thermographically studied with N.S. Kurnakov's pyrometer and differential thermocouple. The  $\text{K}_2\text{SO}_4$  amount was 50, 100, 150, 200, and 250% of the theoretical amount calculated from  $(\text{Li,Na})_2\text{O} \cdot \text{Al}_2\text{O}_3 \cdot 4\text{SiO}_2 + \text{K}_2\text{SO}_4 = (\text{Li,Na})\text{SO}_4 + \text{K}_2\text{O} \cdot \text{Al}_2\text{O}_3 \cdot 4\text{SiO}_2$ , and 20, 40, 60, 80, and 100% of the mineral weight (10-20 g of  $\beta$ -spodumene). Part of all mixtures was heated to 1000-1100°C and measured pyrometrically, another part was annealed in corundum crucibles in a muffle furnace for 3 hr at a constant temperature of 700, 800, 900, 950, 1000, 1050, and 1100°C. Part

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of these masses was heated to 1000-1100°C and thermographically studied. Another part was extracted with water, and the residues were heated to 850-1050°C and studied thermographically, in part also by X-ray phase analysis. Heating rate of mixtures and sintering masses: 8-12°C/min; of residues: 16-18°C/min. The phase transformation ranges were determined as intervals between initial and final temperatures of the endothermic effects. All sintering masses made in different ways may form a five-component system in the simplest case:  $K_2SO_4$ ,  $Na_2SO_4$ ,  $Li_2SO_4$ ,  $\beta$ -spodumene, and newly formed conversion product. The first three soluble substances are likely to determine the phase transformations and thermal effects. When studying the liquidus of the system, Ye.K. Akopov and A.G. Bergman showed that the diagram consisted of 10 crystallization fields. The largest is the field of solid solutions  $(Na,K)_2SO_4$  with monovariant rupture curves. The solidus is even more complicated since decomposition into components with polymorphous modification, e.g.,  $Li_2SO_4$ , occurs at a drop in temperature. The heating curves of the mechanical mixtures are identical and have two thermal effects: the first corresponds to the liquidus  $Li_2SO_4$ .

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-  $\text{Na}_2\text{SO}_4$ - $\text{K}_2\text{SO}_4$  in the range of almost pure  $\text{K}_2\text{SO}_4$ , the second corresponds to the reversible, polymorphous  $\text{K}_2\text{SO}_4$  conversion. The temperature range of the first effect (968-998°C) is similar for all curves since an increase in the  $\text{K}_2\text{SO}_4$  content (viscous component) increases the Li- and Na-transformation of  $\beta$ -spodumene into the salt system. It was found that  $\beta$ -spodumene reacted with  $\text{K}_2\text{SO}_4$  already below 900°C since solid solutions were already formed in the system  $\text{Li}_2\text{SO}_4$ - $\text{Na}_2\text{SO}_4$ - $\text{K}_2\text{SO}_4$  before the thermal effect set in.

The heating curves of cakes at 700°C are also identical. As here a reaction occurred already in the first sintering process at 700°C, the first effect lies at 939-976°C. The heating curves of cakes sintered at 800°C also show a third effect between 403 and 432°C of the transformation in the solidus range of the ternary salt system. The heating curves of 900, 1000, and 1100°C cakes have much in common (Fig. 1). The transformation range corresponding to the liquidus of the ternary system lies above 900°C only at cake compositions 5/1 and 5/2; at compositions 5/3, 5/4, 1/1, however, it lies between 650 and 700°C, since they do not correspond to the crystalli-

Card 4/10

Study of the reaction of ...

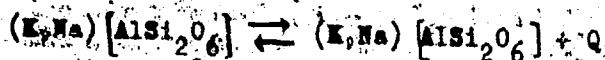
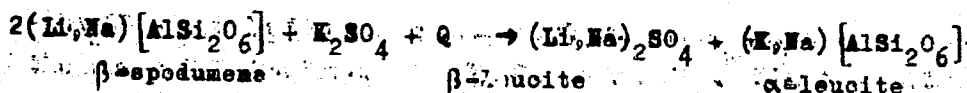
S/153/61/004/001/005/009  
B110/B203

zation field of solid solutions of  $\text{Li}_2\text{SO}_4\text{-Na}_2\text{SO}_4\text{-K}_2\text{SO}_4$  (A), but to that of the compound  $\text{Li}_2\text{SO}_4\text{-K}_2\text{SO}_4$ . The 2nd and 3rd ranges correspond to processes in system (A) in solid state, the 4th transformation range of 22-650°C (Table 2) at compositions 5/4 and 1/1, is also observed in unsoluble residues. The reversible thermal effect of residues corresponds to that of polymorphous conversion of polymorphous leucite. Thus, leucite is formed by polymorphous enantiotropic conversion in the reaction of  $\beta$ -spodumene with  $\text{K}_2\text{SO}_4$ . To prove this,  $\beta$ -spodumene was again sintered with  $\text{K}_2\text{SO}_4$  (1/1) at 1100°C for 3 hr. The dried residue annealed at 200°C was monophasic with an average  $n = 1.508 \pm 0.003$ .  $\text{SiO}_2$  was separated on addition of HCl. A silicate analysis yielded (in %):  $\text{K}_2\text{O} = 21.40$ ;  $\text{Al}_2\text{O}_3 = 23.51$ ;  $\text{SiO}_2 = 55.17$ . The X-ray phase analysis of the residues of cakes sintered at 700-800°C (ratio 1:1) yielded unchanged spodumene; that of the residue of cakes sintered above 900°C yielded leucite. The reactions

Card 5/10

Study of the reaction of . . .

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B110/B203



start at 700°C in the solid phase, and increase at 900°C with increasing K<sub>2</sub>SO<sub>4</sub> consumption. Heating to 1100°C leads to a quantitative exchange. A temperature above 1100°C reduce again the water-soluble Li fraction. There are 2 figures, 2 tables, and 14 references: 11 Soviet-bloc and 3 non-Soviet-bloc.

ASSOCIATION: Moskovskiy institut tonkoy khimicheskoy tekhnologii im. M.V. Lomonosova, Kafedra tekhnologii redkikh i rasseyannykh elementov (Moscow Institute of Fine Chemical Technology imeni M.V. Lomonosov, Department of Technology of Rare and Dissipated Elements)

SUBMITTED: May 9, 1959.

Card 6/10

PLYUSHCHEV, V.Ye.

Reactions of minerals containing rare alkali elements with salts and oxides in the process of fusion and sintering. Part 5: Mechanism of the fusion of  $\alpha$ -spodumene with potassium sulfate. Izv. vys. ucheb. zav.; khim. i khim. tekhn. 4 no. 2:265-270 '61.  
(MIRA 14:5)

1. Moskovskiy institut tonkoy khimicheskoy tekhnologii im. M.V. Lomonosova. Kafedra tekhnologii redkikh i rasseyannykh elementov.  
(Spodumene) (Potassium sulfate)

STEPIN, B.D.; PLYUSHCHEV, V.Ye.

Applicability limits for the method of additions in the flame photometric determination of alkaline elements. Izv.vys.uche. zav.; khim. i khim.tekh. 4 no.4:569-573 '61. (IRA 15:1)

1. Moskovskiy institut tonkoy khimicheskoy tekhnologii imeni Lomonosova, kafedra khimii i tekhnologii redkikh i rasseyannykh elementov.

(Alkali metals--Analysis) (Flame photometry)

S/153/61/004/006/005/008  
E111/E453

AUTHOR: Plyushchev, V.Ye.

TITLE: Investigation of the reaction of minerals, containing rare alkali elements, with salts and oxides during sintering and fusion. VII. Study of the decomposition of spodumene with calcium chloride

PERIODICAL: Izvestiya vysshikh uchebnykh zavedeniy.  
Khimiya i khimicheskaya tekhnologiya, v.4, no.6, 1961,  
1011-1015

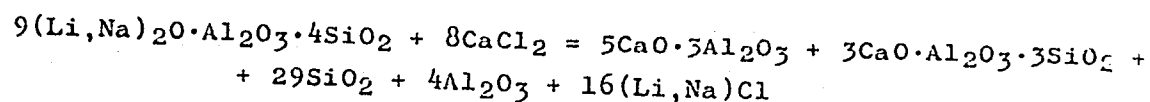
TEXT: There are very few methods for the decomposition of lithium minerals with the chlorides of the alkaline and alkaline-earth elements at high temperatures. Calcium chloride can be used for both lepidolite and spodumene. In the present work the author reports experiments with calcium chloride and beta-spodumene. The sample used contained 5.44%  $\text{Li}_2\text{O}$  and 3.20%  $\text{Na}_2\text{O}$  and its characteristics have been described in detail previously. The reaction was studied with the aid of thermal and X-ray analysis. A sintered (or re-solidified melt) composition of  
Card 1/3



Investigation of the reaction ...

S/153/61/004/006/005/008  
E111/E453

spodumene and anhydrous calcium chloride (obtained by heating together at 750 to 1200°C) was used, as well as the insoluble residues obtained from it by washing with water and drying at 150°C. An excess of calcium chloride was used. X-ray diffraction patterns were obtained from all insoluble residues, using a camera of about 57.3 mm diameter and unfiltered iron radiation at 35 kV and 10 mA. Detailed phase analysis was carried out on the residue from a product obtained at 1200°C with a 1:1 composition and this was used as a basis for similar analyses of other residues. It was concluded that the probable reaction is



The water soluble products of the reaction are  $\text{CaCl}_2$ ,  $\text{NaCl}$  and  $\text{LiCl}$ . The distribution of the lithium chloride between the sintered and gaseous reaction products depends on the temperature at which the process is effected. There is 1 table.  
Card 2/3

Investigation of the reaction ...

S/153/61/004/006/005/008  
E111/E453

ASSOCIATION: Moskovskiy institut tonkoy khimicheskoy  
tekhnologii im. M.V.Lomonosova. Kafedra  
tekhnologii redkikh i rasseyannykh elementov  
(Moscow Institute for Fine Chemical Technology  
imeni M.V.Lomonosov, Department of the Technology of  
Rare and Scattered Elements)

SUBMITTED: January 25, 1960

Card 3/3

S/078/61/006/002/013/017  
B017/B054

AUTHORS: Stepin, B. D., Plyushchev, V. Ye.

TITLE: Coprecipitation of Microquantities of Potassium With Crystalline Precipitates of Rubidium Salts

PERIODICAL: Zhurnal neorganicheskoy khimii, 1961, Vol. 6, No. 2, pp. 462-468

TEXT: The authors studied the distribution of potassium in crystalline precipitates of permanganate, tetraoxalate, rubidium alum, and nickel rubidium picromerite. The initial salts were purified by twofold crystallization. Potassium was determined flame-photometrically with the WCN-51 (ISP-51) spectrograph. The distribution coefficients were calculated by the

formulas  $D_{(K)/Rb} = \frac{x(b-y)}{y(a-x)}$  and  $\lambda_{(K)/Rb} = \frac{\log a(b-y)}{\log b(a-x)}$ , where D = Khlopin constant,  $\lambda_{(K)/Rb}$  = Derner-Hoskins constant, a, b = corresponding micro- and

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Coprecipitation of Microquantities of  
Potassium With Crystalline Precipitates  
of Rubidium Salts

S/078/61/006/002/013/017  
B017/B054

macroquantities of components in the solution before crystallization,  $x, y$  - corresponding amounts of ~~micro-~~ and macrocomponents in the precipitate,  $a-x, b-y$  - corresponding amounts of micro- and macrocomponents in the solution after crystallization. The distribution coefficients of the systems  $\text{KMnO}_4 - \text{RbMnO}_4 - \text{H}_2\text{O}$ ;  $\text{KH}_3(\text{C}_2\text{O}_4)_2 - \text{RbH}_3(\text{C}_2\text{O}_4)_2 - \text{H}_2\text{O}$ ;  $\text{K}_2\text{SO}_4 \cdot \text{Al}_2(\text{SO}_4)_3 - \text{Rb}_2\text{SO}_4 \cdot \text{Al}_2(\text{SO}_4)_3 - \text{H}_2\text{O}$ , and  $\text{K}_2\text{SO}_4 \cdot \text{NiSO}_4 - \text{Rb}_2\text{SO}_4 \cdot \text{NiSO}_4 - \text{H}_2\text{O}$  were determined. It was found that the constants depended on temperature. The fractional crystallization of permanganate and alum was found to be suitable for the production of purest rubidium salts. The separation of rubidium and potassium by fractional crystallization of tetraoxalates is impossible in principle. Purification of rubidium alums from potassium is best made from saturated solutions at 40 - 50°C. A K-enriched solid phase is formed on the surface at higher temperature. Rubidium can be separated from potassium by fractionating the double sulfates of rubidium and nickel at a temperature below 40 - 50°C. M. D. Delepin and G. I. Gorshteyn are

Card 2/3

Coprecipitation of Microquantities of  
Potassium With Crystalline Precipitates  
of Rubidium Salts

S/078/61/006/002/013/017  
B017/B054

mentioned. There are 6 tables and 24 references: 9 Soviet, 4 US, 1 British,  
5 German, and 2 Japanese.

SUBMITTED: October 13, 1959

Card 3/3

✓

STEPIN, B.D.; TIKHONOVA, Ye.A.; PLYUSHCHEV; V.Ye.

Preparation of rubidium hexadecachlorotriantimonide and its  
coprecipitation with potassium chloride. Zhur.neorg.khim. 6  
no.4:890-896 Ap '61. (MIRA 14:4)

1. Moskovskiy institut tonkoy khimicheskoy tekhnologii imeni  
M.V.Lomonosova, Kafedra tekhnologii redkikh i rasseyannykh  
elementov.

(Rubidium compounds)

BOL'SHAKOV, K.A.; POKROVSKIY, B.I.; PLYUSHCHIEV, V.Ya.

Binary systems constituted by alkali nitrates. Zhur.neorg.khim.  
6 no.9:2120-2125 S '61. (MIRA 14:9)  
(Alkali metal nitrates) (Systems (Chemistry))

26285  
S/078/61/006/009/007/010  
B127/B101

5.2100

AUTHORS:

Samuseva R. G., Plyushchev, V. Ye.

TITLE:

The fusibility of binary cesium and sodium halide systems

PERIODICAL:

Zhurnal neorganicheskoy khimii, v. 6, no. 9, 1961, 2139-2141

There is a need for study of sodium and cesium halides is highly interesting and only the system NaCl-CsCl has been investigated at present. The halides of these metals have been studied in detail. The authors investigated NaF-CsF, NaBr-CsBr, NaI-CsI, and also NaCl-CsCl which is eutectic (439°C mole % of NaCl and 439°C). The system was studied by means of Kurnakov's pyrometer and heating curves. Cooling curves were used for comparison. The results of a thermal analysis are given in tables. All systems are eutectic: NaCl-CsCl: 34.5 mole % of NaCl and 490°C; NaBr-CsBr: 37.5 mole % of NaBr and 460°C; NaI-CsI: 45 mole % of NaI and 435°C; NaF-CsF: 20 mole % of NaF and 615°C. There are 4 figures, 4 tables, and 3 Soviet-bloc references.

Card 1/6



26286  
S/078/61/006/009/007/010  
B127/E101

The fusibility of binary cesium...

ASSOCIATION: Moskovskiy institut tonkoy khimicheskoy tekhnologii im.  
M. V. Lomonosova (Moscow Institute of Fine Chemical  
Technology imeni M. V. Lomonosova)

SUBMITTED: July 26, 1960

Legend to the Tables: (1) Results of thermal analysis of the system ....  
(2) temperature of critical point, °C; (3) liquidus; (4) solidus; (5)  
polymorphous conversion of CsCl.

Card 2/6

15.2230

27897

S/078/61/006/010/004/010

B121/B101

AUTHORS: Grizik, A. A., Plyushchev, V. Ye.

TITLE: Lithium meta-zirconate and lithium meta-hafnate

PERIODICAL: Zhurnal neorganicheskoy khimii, v. 6, no. 10, 1961, 2249-2253

TEXT: A method for preparing lithium meta-zirconate and lithium meta-hafnate has previously been described (B. I. 12: A. A. Grizik and V. Ye. Plyushchev: Zh. Vsesoyuzn. khim. obshch-va im. Mendeleeva 5, 349 (1960)). Some properties of these compounds are discussed in the present paper. High-purity  $ZrO_2$  (containing 0.03 % of  $HfO_2$ ),  $HfO_2$  (containing less than 1 % of  $ZrO_2$ ), and chemically pure lithium carbonate were used to prepare the zirconate and hafnate. The initial components ( $LiO_2:MeO_2=1:1$ ) were thoroughly ground and then sintered in an alundum crucible at  $1100^\circ C$  for 1 1/2 hr. The Debye patterns were photographed by means of an PKA (RKD) camera with ECB (BSV) tube and measured with an M3A-5 (IZA-5) comparator. The crystal density was ascertained pycnometrically at  $20^\circ C$

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S/078/61/006/010/004/010  
B121/B101

Lithium meta-zirconate and lithium...

and found to be:  $\text{Li}_2\text{ZrO}_3$ :  $4.123 \pm 0.003$ ;  $\text{Li}_2\text{HfO}_3$ :  $6.453 \pm 0.003$ . The melting points of the compounds were determined with an optical pyrometer in air and found to be:  $\text{Li}_2\text{ZrO}_3$ : 1600-1620;  $\text{Li}_2\text{HfO}_3$ : 1600-1620. These lithium meta-zirconate and meta-hafnate compounds are not hygroscopic. Lithium meta-zirconate is more readily solubilized in water than lithium meta-hafnate. The tests were performed by measuring the pH with an AN-5 (LP-5) potentiometer in buffer solutions. The solubility of lithium meta-zirconate and meta-hafnate in sulfuric and hydrochloric acids was studied. Concentrated hydrochloric and sulfuric acids decompose lithium meta-hafnate. Lithium meta-zirconate and meta-hafnate are stable in alkaline solutions (0.1 - 10 N KOH). There are 3 tables and 17 references: 6 Soviet and 11 non-Soviet. The three most recent references to English-language publications read as follows: J. Schenck, Nucleonics, 10, N8. 54 (1952); W. M. Fenton, Amer. Ceram. Soc. Bull., 27, 492 (1948); C. E. Curtis et al., J. Amer. Ceram. Soc., 27, 458 (1954).

SUBMITTED: August 10, 1960

Card 2/2

S/020/61/136/002/020/034  
B016/B060

AUTHORS: Komissarova, L. N., Yuranova, L. I., and Plyushchev, V. Ye.

TITLE: Synthesis and Thermal Stability of Dihydrates of Oxy-nitrates of Zirconium and Hafnium

PERIODICAL: Doklady Akademii nauk SSSR, 1961, Vol. 136, No. 2, pp. 350-353

TEXT: A study has been made of the synthesis of zirconium and hafnium oxy-nitrate dihydrates and their thermal stability. While data available in the literature on the former dihydrate are insufficient and contradictory, the latter is as yet undescribed. Zirconium (hafnium-) oxy-chloride octohydrate, which contains a constant amount of crystal water, was proved to be the only usable initial substance for the synthesis of these compounds. A weighed-in portion of these salts was treated with 100%  $\text{HNO}_3$  ( $\text{MeO}_2 : \text{HNO}_3 = 1 : 3$  to  $1 : 6$ ).  $1 : 4.5$  was found as the optimum ratio. All was blown through the solution until the yellow color disappeared, and at each blow with a pipette, on the basis of the analysis.

Card 1/2

Synthesis and Thermal Stability of Dihydrates  
of Oxy-nitrates of Zirconium and Hafnium

S/020/61/136/002/020/034  
B016/B060

the following formulas are ascribed to the compounds synthesized:  
 $ZrO(NO_3)_2 \cdot 2H_2O$  and  $HfO(NO_3)_2 \cdot 2H_2O$ . They are white crystalline substances.

The data obtained for equilibrium displayed certain differences between the zirconium and hafnium compounds regarding the stability and decomposition on heat treatment. They are, however, both thermally unstable and decompose completely at 400°C, when monoclinic  $ZrO_2$  results from

zirconium oxy-nitrate dihydrate with a 54.1% loss of weight. The hafnium compound displays a greater thermal stability, especially the monohydrate forming in between. Both substances decompose stepwise (the stages are less marked in the zirconium compound). There are 2 figures, 2 tables, and 9 non-Soviet references: 3 French, 1 British, and 4 German.

ASSOCIATION: Moskovskiy institut tonkoy khimicheskoy tekhnologii im. M. V. Lomonosova (Moscow Institute of Fine Chemical Technology imeni M. V. Lomonosov)

PRESENTED: June 24, 1960, by V. I. Spitsyn, Academician

SUBMITTED: June 22, 1960

Card 2/2

29018

TITLE:

New data on the behavior of zirconium and hafnium oxynitrates in aqueous solutions and organic solvents

PERIODICAL: Akademiya nauk SSSR. Doklady, v. 140, no. 4, 1961, 855-858

TEXT: Almost all studies described in the literature with regard to the chemistry of zirconium nitrate solutions concerned microquantities of zirconium and highly diluted solutions (Ref. 3: V. I. Paramonova, ZhNKh, 1, 1905 (1956); Refs. 4-6, see below). In practice, the behavior of zirconium and hafnium oxynitrates in more concentrated solutions is interesting, particularly when studying extraction methods for separating zirconium from hafnium. Therefore, the authors investigated the dependence of pH value, specific electrical conductivity, density, and viscosity of zirconium and hafnium oxynitrate solutions on the concentrations of these compound in aqueous solutions. Moreover, the solubility of oxynitrates in organic solvents was studied. The compounds  $\text{ZrO}(\text{NO}_3)_2 \cdot 2\text{H}_2\text{O}$ ,  $\text{ZrO}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ , and  $\text{HfO}(\text{NO}_3)_2 \cdot 2\text{H}_2\text{O}$ ,  $\text{HfO}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$  were

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29018  
S/020/61/140/004/019/023  
B106/B110

New data on the behavior of ...

tested. Figs. 1 and 2 show results obtained for pH value and specific electrical conductivity of aqueous solutions of these compounds. Since the hydrolysis of oxynitrates decreases with increasing concentration, the decrease of the pH value is only due to the increasing absolute quantity of dissolved salts. Hydrolysis of zirconium and hafnium oxynitrates in aqueous solutions was found to depend on time. The state of equilibrium is attained only two weeks after the solutions have been prepared. A rise in temperature effects stronger hydrolysis. As expected, oxynitrates of hafnium proved to be stronger bases than those of zirconium. The density and viscosity values measured for aqueous solutions of the oxynitrates concerned are shown in Figs. 3 and 4. 15 organic compounds of different classes were selected to study the solubility of zirconium and hafnium oxynitrates in organic solvents. Measurements were made at 20 and 40°C. Temperature variations were found to affect solubility in a significant manner. It is evident that oxynitrates are more soluble in polar solvents, only slightly, or not at all, in aprotic solvents. The compounds studied are unsoluble in acetophenone, anisole, and benzyl ethers, chloroform, carbon tetrachloride, and dichloro ethane. The solubility of oxynitrates decreases with increasing chain length and

29018-

S/020/61/140/004/019/023  
B106/B110

New data on the behavior of ...

branching of the saturated alcohols used as solvents. Under equal conditions, oxynitrates of zirconium are better soluble in organic solvents than those of hafnium. There are 4 figures, 5 tables, and 7 references: 4 Soviet and 3 non-Soviet. The three references to English-language publications read as follows: Ref. 4: B. Lister, L. McDonald, J. Chem. Soc., 1952, 4315; Ref. 5: R. Connick, W. McVey, J. Am. Chem. Soc., 71, 3182 (1949); Ref. 6: R. Connick, N. McVey, J. Am. Chem. Soc., 73, 1171 (1951).

ASSOCIATION: Moskovskiy institut tonkoy khimicheskoy tekhnologii im. M. V. Lomonosova (Moscow Institute of Fine Chemical Technology imeni M. V. Lomonosov)

PRESENTED: April 25, 1961, by V. I. Spitsyn, Academician

SUBMITTED: April 22, 1961

Card 3/8



AUTHORS:

Stetsko, R. M., *Khimicheskaya promyshlennost'*, no. 6, 1962, 18 - 22

TITLE:

Extractive separation of rubidium and potassium

PERIODICAL:

*Khimicheskaya promyshlennost'*, no. 6, 1962, 18 - 22

TEXT: The distribution of organic K and Rb compounds is studied between the aqueous phase and alcohols, ketones and esters of  $C_2-C_5$  monocarboxylic acids.. Rb and K carbonates obtained from  $RbCl$  and  $K_2CO_3$  respectively, were dissolved in  $H_2O$  and heated to  $50 - 60^\circ C$  while stirring; organic acid was added up to a pH of  $6 - 6.5$ , then excess acid ( $2 - 4$  ml) was added, the solution boiled down, and the residue mixed with acetone. Extraction was performed in separating funnels. The solvent is saturated with  $H_2O$  before being used for the extraction so that equilibrium is established exclusively by the organic solvent and the test substance. The salts are converted to sulfates to determine their content in both phases. The calculated separation coefficient,  $\beta$ , agrees with the usual values, but may be changed significantly in the presence of two 1-anion

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33280  
S/078/62/007/002/010/019  
B119/B110

S 2100

AUTHORS: Stepin, B. D., Plyushchev, V. Ye.

TITLE: Some properties of rubidium iodine chlorobromide and its  
cocrystallization with potassium

PERIODICAL: Zhurnal neorganicheskoy khimii, v. 7, no. 2, 1962, 394 - 400

Abstract: The study of the properties of  $Rb[I(Cl Br)]$  is of interest for the  
preparation of solid-state electrolytes. Rubidium iodine chlorobromide  
crystals were obtained by the reaction of rubidium iodide with  
hydrogen bromide and hydrogen chloride. The composition of the  
crystals was determined by flame photometry. The content of  
YM-2 (UM-2) monochromator with photomultiplier could not be used) was  
verted to  $Rb[I(Cl Br)]$ . (ГОСТ 4159-48 (GOST 4159-48) I powder was mixed  
with (ГОСТ 4109-48 (GOST 4109-48) Br and after removal of the excess Br  
in  $N_2$  flow the concentrated  $RbCl$  solution was added under continuous  
stirring at 70 - 80°C. The  $Rb[I(Cl Br)]$  formed was precipitated as fine  
orange-coloured crystals by cooling to ~0°C. The product was studied as  
to: K content (flame photometry), content of halogens (potentiometric

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S/078/62/007/002/010/019  
B119/B110

Some properties of rubidium...

titration), hydrate water (titration with Fischer's reagent), solubility, and differential thermogram (in the pyrograph of F. V. Syromyatnikov (Ref. 12: Avt. svid. SSSR No. 62899 (1943)). УПМ ("UPI") fluorized oil was used as sealing liquid. Results: Solubility of  $\text{Rb}[\text{I}(\text{Cl Br})]$  in water at  $+0.1^\circ\text{C}$ : 34.7% by weight; at  $70^\circ\text{C}$ : 87.4% by weight. Analytically found formula of the product:  $\text{Rb}[\text{I}(\text{Cl Br})] \cdot \text{H}_2\text{O}$ . Melting point  $207.5 - 209.0^\circ\text{C}$ . (After dehydration at  $130^\circ\text{C}$ : melting point  $216 - 218^\circ\text{C}$ ). The thermogram of  $\text{Rb}[\text{I}(\text{Cl Br})]$  shows a small endothermic effect at  $112.5^\circ\text{C}$  and a strong effect at  $231.5^\circ\text{C}$  (corresponding  $\text{Rb}[\text{I}(\text{Cl Br})] \rightleftharpoons \text{RbCl} + \text{IBr}$ ). Cocrystallization of K: A K content of 10.02% in the initial  $\text{RbCl}$  decreases (after thermal decomposition of the  $\text{Rb}[\text{I}(\text{Cl Br})]$  precipitated) to 0.04% in the final  $\text{RbCl}$ . An initial K content of 0.02% decreases to 0.0005%. In the presence of acetic acid in the reaction mixture ( $\text{pH} = 2.0 - 2.7$ ) the K content in the final product ( $\text{RbCl}$ ) is still further reduced (from 0.02 - 0.05% to 0.0002%). Thus,  $\text{RbCl}$  is purified from Li and Na to the same extent; whereas Cs is enriched. V. G. Khlopin is mentioned. There are 4 figures, 5 tables, and 16 references: 9 Soviet and 7 non-Soviet. The four most recent references to English-language publications read as follows: H. Wells, H. W. Wheeler. Amer. J. Sci., 43,

Card 2/3

APPROVED FOR RELEASE: 07/13/2001

CIA-RDP86-00513R001341410001-6"

Some properties of rubidium...

S/078/62/007/002/010/019  
B119/B110

475 (1892); I. M. Kolthoff, H. Yutzu. Ind. Eng. Chem. Anal. Ed., 2, 75 (1937); H. W. Cremer, D. R. Duncan. J. Chem. Soc., 1857 (1931); H. W. Cremer, D. R. Duncan. J. Chem. Soc., 2031 (1932).

SUBMITTED: February 27, 1961

Card 3/3

37169

S/078/62/007/005/008/014

B101/B110

21.2000

AUTHORS: Grizik, A. A., Plyushchev, V. Ye.

TITLE: Synthesis and properties of sodium zirconate and hafnate

PERIODICAL: Zhurnal neorganicheskoy khimii, v. 7, no. 5, 1962, 1054-1061

TEXT: To study the formation of compounds in the system  $\text{Na}_2\text{O} - \text{ZrO}_2$  the reaction of  $\text{Na}_2\text{CO}_3$  with  $\text{ZrO}_2$  between 800 and 1400°C was investigated, the  $\text{Na}_2\text{CO}_3/\text{ZrO}_2$  ratio being varied between 1:4 and 4:1. The mixtures of the initial substances powdered to 200 mesh were pressed into tablets and heated. Only the compound  $\text{Na}_2\text{ZrO}_3$  was found to form, which was detected by Debye pattern after 1-hr keeping at 800°C. The  $\text{Na}_2\text{O}$  sublimation observed confirmed a direct reaction in the system  $\text{Na}_2\text{O} - \text{ZrO}_2$ . The use of  $\text{Na}_2\text{CO}_3$  yielded no pure metazirconates. At too low a temperature, the product contained  $\text{Na}_2\text{O}$ , at too high a temperature, the metazirconate

Card 1/3

Synthesis and properties of sodium ...

S/078/62/007/005/008/014  
B101/B110

decomposed. Thus, experiments with  $\text{NaNO}_3$  were made, and the following optimum conditions were found for the production of coarse-crystalline  $\text{Na}_2\text{ZrO}_3$ : heating of  $\text{Na}_2\text{CO}_3 + \text{ZrO}_2$ , ratio 1.5 : 1, at 1200 - 1300°C, addition of 10%  $\text{NaNO}_3$ , and short heating at 1000°C. The small content of  $\text{Na}_2\text{O}$  can be washed out with ethylene glycol or absolute alcohol.  $\text{Na}_2\text{HfO}_3$  was prepared analogously. Both compounds show birefringence. The mean refractive indices are: 1.76 for  $\text{Na}_2\text{ZrO}_3$ , 1.78 for  $\text{Na}_2\text{HfO}_3$ ; X

$d_{\text{Na}_2\text{ZrO}_3}^{20} = 4.060 \pm 0.003$ ;  $d_{\text{Na}_2\text{HfO}_3}^{20} = 5.763 \pm 0.005$ . The interplanar spaces of the two compounds were determined by Debye patterns. Both compounds hydrolyze quickly and completely in water at 100°C, but only slowly at 20°C, with formation of polyzirconates which are being studied. In air, the compounds  $\text{Na}_2\text{Me}^{\text{IV}}\text{O}_3$  decompose slowly owing to reaction with water vapor and  $\text{CO}_2$ , the metazirconate being more stable than the metahafnate. After 30 min  
Card 2/3

37174

S/078/62/007/005/014/014  
B101/B110

18.9200

AUTHORS: Samuseva, R. G., Poletayev, I. F., Plyushchev, V. Ye.

TITLE: Study of the fusibility in the systems  $\text{Na}_2\text{Cr}_2\text{O}_7$  -  $\text{Rb}_2\text{Cr}_2\text{O}_7$   
and  $\text{Na}_2\text{Cr}_2\text{O}_7$  -  $\text{Cs}_2\text{Cr}_2\text{O}_7$

PERIODICAL: Zhurnal neorganicheskoy khimii, v. 7, no. 5, 1962, 1146-1149

TEXT: The melting-point diagrams of the systems  $\text{Na}_2\text{Cr}_2\text{O}_7$  -  $\text{Rb}_2\text{Cr}_2\text{O}_7$  (I) and  $\text{Na}_2\text{Cr}_2\text{O}_7$  -  $\text{Cs}_2\text{Cr}_2\text{O}_7$  (II) were plotted by means of thermal analysis to extend the applicability of Rb and Cs compounds. The compound  $\text{Na}_2\text{Cr}_2\text{O}_7 \cdot \text{Rb}_2\text{Cr}_2\text{O}_7$ , m. p.  $339^\circ\text{C}$ , is formed in system I; it forms eutectics with the components of the system:  $\text{Na}_2\text{Cr}_2\text{O}_7 \cdot \text{Rb}_2\text{Cr}_2\text{O}_7 + \text{Na}_2\text{Cr}_2\text{O}_7$ , m. p.  $303^\circ\text{C}$ , contains 22 mole%  $\text{Rb}_2\text{Cr}_2\text{O}_7$ ;  $\text{Na}_2\text{Cr}_2\text{O}_7 + \text{Rb}_2\text{Cr}_2\text{O}_7$ , m. p.  $317^\circ\text{C}$ , contains 75 mole%  $\text{Rb}_2\text{Cr}_2\text{O}_7$ . A polymorphous conversion of the sodium bichromate was observed at  $248^\circ\text{C}$ . The low thermal effect of this conversion  
Card 1/2

S/078/62/007/006/016/024  
B119/B138

AUTHORS: Samuseva, R. G., Plyushchev, V. Ye., Yegorova, R. S.  
TITLE: Joint solubility of cesium and sodium iodides  
PERIODICAL: Zhurnal neorganicheskoy khimii, v. 7, no. 6, 1962, 1415-1417  
TEXT: The system NaI - CsI - H<sub>2</sub>O was investigated at 25 and 50°C (isotherm). The solubility polytherm of the system CsI - H<sub>2</sub>O was studied between 0 and 70°C. The solubility of CsI in H<sub>2</sub>O is 27.60% by weight at 0°C and 64.95% by weight at 70°C. The figure shows the solubility isotherms at 25 and 50°C. In the system NaI - CsI - H<sub>2</sub>O, NaI has a very strong salting-out effect on CsI. There are 1 figure and 2 tables. The most important English-language references are: A. Hill, H. Willson, I. Bishop. J. Amer. Chem. Soc., 55, 521 (1933); H. W. Foote. Amer. Chem. J., 29, 207 (1903).  
ASSOCIATION: Moskovskiy institut tonkoy khimicheskoy tekhnologii im. M. V. Lomonosova (Moscow Institute of Fine Chemical Technology imeni M. V. Lomonosov)  
Card 1/1 ✓

SAMUSEVA, R.G.; YEGOROVA, R.S.; PLYUSHCHEV, V.Ye.

Ternary system sodium bromide - cesium bromide - water. *Zhur.neorg.khim.*  
7 no.7:1666-1669 JI '62. (MIRA 16:3)

1. Moskovskiy institut tonkoy khimicheskoy tekhnologii imeni Lomonosova.  
(Sodium bromide) (Cesium bromide)



GRIZIK, A.A.; PLYUSHCHEV, V.Ye.; PLESKOVA, I.A.

Synthesis and some properties of potassium dizirconate.. Zhur.  
neorg.khim. 7 no.12:2702-2708 D '62. (MIRA 16:2)  
(Potassium zirconate)

37386

S/020/62/143/006/019/024  
B106/B138

11.4100  
AUTHORS:

Plyushchev, V. Ye., Stepina, S. B., Stepin, B. D., and  
Lepeshkova, L. I.

TITLE:

Heterotripolyhalides of alkali elements with similar properties and their importance for the production of pure rubidium and cesium compounds

PERIODICAL: Akademiya nauk SSSR. Doklady, v. 143, no. 6, 1962, 1364-1367

TEXT: The possibility of producing pure Rb and Cs compounds via complex heterotripolyhalides is thoroughly discussed with the aid of 27 references. A method developed by the authors (V. Ye. Plyushchev, B. D. Stepin, Author's certificate USSR no. 132627 (1960); B. D. Stepin, V. Ye. Plyushchev, Author's certificate USSR no. 140051 (1961)) provides for the production of Rb preparations containing only 0.0002% potassium, from industrial RbCl containing 2 - 3% K. Rb preparations of such high purity had not been obtained by methods described before. In the present simple and economic procedure, RbCl is twice (first in aqueous solution, then in 0.5 M acetic acid) converted at 90°C into the complex  $\text{Rb}[\text{I}(\text{ClBr})] \cdot \text{H}_2\text{O}$ .

Card 1/3

SHKLOVER, L.R.; PLYUSHCHEV, V.Ye.

Thermographic characteristics of o-phthalic acid and its derivatives. Izv.vys.ucheb.zav.; khim. i khim. tekhn. 6 no.6: 957-959 '63. (MIRA 17:4)

1. Moskovskiy institut tonkoy khimicheskoy tekhnologii imeni Lomonosova, kafedra tekhnologii redkikh i rasseyannykh elementov.

PLYUSHCHEV, V.Ye.; AMOSOV, V.M.; VARFOLOMEYEV, M.B.

Synthesis and some properties of lower crystal hydrates of yttrium, lanthanum, and lanthanum series perrhenates. Dokl. AN SSSR 150 no.1:105-108 My '63. (MIRA 16:6)

1. Moskovskiy institut tonkoy khimicheskoy tekhnologii im. M.V. Lomonosova. Predstavleno akademikom I.V.Tananayevym.  
(Rare earth compounds) (Perrhenic acid)

SHAKHNO, I.V.; PLYUSHCHEV, V.Ye.; TITUNINA, Ye.M.; SAMUSEVA, R.G.

Solubility in the system  $\text{Na}_2\text{CrO}_4$  -  $\text{Cs}_2\text{CrO}_4$  -  $\text{H}_2\text{O}$  at 25 and  
50°C. Zhur. neorg. khim. 8 no.6:1466-1469 Je '63.  
(MIRA 16:6)

(Alkali metal chromates)  
(Solubility)

SAMUSEVA, R.G.; PLYUSHCHEV, V.Ye.; POLETAYEV, I.F.

Phase diagrams of the systems  $\text{Na}_2\text{CrO}_4$  -  $\text{Rb}_2\text{CrO}_4$  and  
 $\text{Na}_2\text{CrO}_4$  -  $\text{Cs}_2\text{CrO}_4$ . Zhur.neorg.khim.<sup>48</sup> no.1:167-171 Ja '63.  
(MIRA 16:5)

1. Moskovskiy institut tonkoy khimicheskoy tekhnologii imeni  
M.V.Lomonosova.

(Alkali metal chromates) (Thermal analysis)

PLYUSHCHEV, V.Ye.; STEPINA, S.B.; LEPESHKOVA, L.I.

Iso(tri)polyhalides very similar in properties to alkaline metals, and their use in the removal of cesium from rubidium compounds. Dokl. AN SSSR 148 no.3:601-604 Ja '63. (MIRA 16'2)

1. Moskovskiy institut tonkoy khimicheskoy tekhnologii im. M.V. Lomonosova. Predstavleno akademikom I.V. Tananayevym.  
(Halides) (Rubidium compounds) (Cesium)

KOMISSAROVA, L.N.; SAVEL'YEVA, M.V.; PIYUSHCHEV, V.Ye.

New zirconium and hafnium hydroxyacetates. Zhur.neorg.khim. 8 no.1:  
56-62 Ja '63. (MIRA 16:5)

1. Moskovskiy gosudarstvennyy universitet imeni M.V.Lomonosova i  
Moskovskiy institut tonkoy khimicheskoy tekhnologii imeni  
M.V.Lomonosova.

(Zirconium compounds) (Hafnium compounds) (Glycolic acid)



STEPINA, S.B.; PLYUSHCHEV, V.Ye.; LEPESHKOVA, L.I.

Removal of cesium microimpurities from rubidium salts. Zhur.-  
neorg.khim. 8 no.2:487-489 F '63. (MIRA 16:5)

1. Moskovskiy institut tonkoy khimicheskoy tekhnologii imeni  
M.V.Lomonosova.

(Rubidium salts) (Cesium iodide)

PLYUSHCHEV, V.Ye., doktor khim. nauk, red.; ALEKSEYEV, V.A., red.;  
RYBKINA, V.P., tekhn. red.

[Cesium] TSezii; sbornik statei. Moskva, Izd-vo inostr. lit-  
ry, 1963. 230 p. (MIRA 16:4)  
(Cesium compounds)

I 12707-63 FWP(q)/FMT(m)/BDS AFTTC JD/JG  
 ACCESSION NR: AP3000303 8/0020/6, 150/001/0105/0108

AUTHOR: Plyushchev, V. Ye.; Amosov, V. M.; Varfolomeyev, M. B.

TITLE: The synthesis and several properties of lower crystallohydrates of yttrium, lanthanum and lanthanoid perhenates

SOURCE: AN SSSR. Doklady, v. 150, no. 1, 1963, 105-108

TOPIC TAGS: yttrium, lanthanum, lanthanoid perhenate, lower crystallohydrate

ABSTRACT: The existence of anhydrous perhenates has not been previously established. This article reports a method of preparation of anhydrous perhenates of the rare earth elements by dehydration of their lower crystallohydrates. The obtained perhenates of yttrium, lanthanum and lanthanoids are soluble in water in considerable proportions and thus the described synthesis can utilize the starting materials more effectively with a product yield of 95%. The synthesized perhenates contain a minimum amount of water of crystallization. The rare earth perhenates obtained at 75-80C are non-hygroscopic, fine crystals which readily dissolve in water, alcohol and acetone, and are stable between the temperature interval of 200-550C. Orig. art. has: 1 figure and 1 table.

ASSOCIATION: Moskovskiy institut tonkoy khimicheskoy tekhnologii im. M. V. Card 1/2

1 27181-65 EWT(m)/EPF(c)/EPF(n)-2/EWP(j)/T/EWP(t)/EWP(b)/EWA(c) Pc-l/pr-l/  
Pu-l IJP(c)/RPL JD/WW/JG/RM

ACCESSION NR: AP4009348

S/0078/64/009/001/0125/0127

AUTHOR: Plyushchev, V. Ye.; Shklover, L. P.; Rozdin, I. A.

4 3

3 5

6

TITLE: Synthesis of zirconium and hafnium phthalocyanins 1

27

27

SOURCE: Zhurnal neorganicheskoy khimii, v. 9, no. 1, 1964, 125-127

TOPIC TAGS: zirconium phthalocyanin, zirconium phthalocyanin synthesis, zirconium phthalocyanin purification, zirconium phthalocyanin absorption spectra, hafnium phthalocyanin, hafnium phthalocyanin synthesis, hafnium phthalocyanin purification, hafnium phthalocyanin absorption spectra

ABSTRACT: Zirconium and hafnium phthalocyanins having the composition  $C_{32}H_{15}N_8Cl \cdot Me(OH)_2 \cdot 2H_2O$ , where  $Me = Zr, Hf$ , are prepared by reacting  $\alpha$ -phthalonitrile with the metal tetrachloride (4:1 molar ratio) at 170-190C ( $ZrOCl_2 \cdot 8H_2O$  practically does not react with phthalonitrile). The compounds are stable; the crude pigments can be purified by reprecipitating from the

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L 27101-65

ACCESSION NR: AP4009348

2

from the concentrated  $H_2SO_4$ . Absorption spectra for solutions of Zr and Hf phthalocyanins in alpha-bromonaphthalene in the 400 - 700 millimicron range are presented; the maximum wave length absorption bands are at 693 & 691 respectively. " I. F. Zakharchenko & T. A. Trushina participated in the experimental part of the work." Orig. art. has: 1 figure and 1 table

ASSOCIATION: None

SUBMITTED: 08Jan63

ENCL: 00

SUB CODE: 10, 60

NO REF SOV: 010

OTHER: 006

Card 2/2

PLYUSHCHEV, V.Ye.; SHKLOVER, L.P.

Synthesis of erbium phthalocyanine. Zhur. neorg. khim. 9 no.2:  
335-339 F'64.

Synthesis and purification of samarium and erbium phthalocyanins.  
Ibid.:340-346 (MIRA 17:2)

SHKLOVER, L.P.; PLYUSHCHEV, V.Ye.

Interaction of o-cyanobenzamide with rare-earth metal salts.  
Zhur. neorg. khim. 9 no.8:1830-1832 Ag '64.

Yttrium derivatives of phthalocyanine. Ibid.:2015-2016

(MIRA 17:11)

L 24824-65 EWT(m)/EWP(t)/EWP(b) ~~INT~~(c) JD/JG

ACCESSION NR: AP4046380

S/0020/64/158/003/0664/0867

AUTHOR: Plyushchev, V. Ye.; Varfolomeyev, M. B.

TITLE: Rare earth element and yttrium perrhenate tetrahydrates

SOURCE: AN SSSR, Doklady\*, v. 158, no. 3, 1964, 664-667

TOPIC TAGS: rare earth perrhenate tetrahydrate, yttrium perrhenate tetrahydrate, synthesis, thermal stability, polymorphic transition

ABSTRACT: The preparation and properties of the rare earth element and yttrium perrhenate tetrahydrates was investigated as a continuation of earlier work (V. Ye. Plyushchev, V. M. Amosov, M. B. Varfolomeyev, DAN, 150, 105 (1963)). The crystallization of the filtered perrhenate solutions, prepared as before, was effected by the two methods: removal of crystals from solution evaporated at 75-80C, or evaporation of the solution almost to dryness and precipitation while cooling to 20-25C. Both methods gave non-hygroscopic coarse water-or alcohol-soluble crystals, colored according to the  $Me^{3+}$  ion, containing 4 molecules

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L 24821-65

ACCESSION NR: AP4046380

of water. The densities and refractive indices of the compounds are tabulated. The thermal stability was investigated. Upon isothermal dehydration all the compounds lost  $H_2O$  above  $50^\circ C$ , with the stability increasing in the series from La to Lu. Compounds in the La-Gd series dehydrated to the monohydrates, in the Tb-Lu (and Y) series, to the dihydrates. Thermograms showed endo effects corresponding to the dehydration, to the polymorphic transition (at  $580-620^\circ C$ ) and to the fusion (above  $850^\circ C$ ) of the dehydrated perhenates. Orig. art. has: 4 figures and 1 table

ASSOCIATION: Moskovskiy institut' ionkoy khimicheskoy tekhnologii im. M. V. Lomonosova (Moscow Institute of Fine Chemical Technology)

SUBMITTED: 04May84

ENCL: 00

SUB CODE: GC, IC

NO REF SOV: 004

OTHER: 001

Card 2/2

PLYUSHCHEV, V. Ye.; STEPINA, S.B.; ZAMINA, G.V.; ZHILYAKOV, V.G.

Investigating the interaction of antimony chloride and bromide with corresponding halides close to the properties of alkali elements. Izv. vys. ucheb. zav.; tsvet. met. 7 no. 4:112-116 '64 (MIRA 19:1)

1. Moskovskiy institut tonkoy khimicheskoy tekhnologii, kafedra khimii i tekhnologii redkikh i rasseyannykh elementov.

SHKLOVER, L.P.; PLYUSHCHEV, V.Ye.; ROZDIN, I.A.; NOVIKOVA, N.A.

Synthesis of titanium phthalocyanine. Zhur. neorg. khim.  
9 no.2:478-479 F'64. (MIRA 17:2)

PLYUSHCHEV, V. Ye.; VARFOLOMEYEV, M. B.

Cerium perxhenate and its crystal hydrates. Izv. vys. ucheb. zav.; khim. i kh'm. tekhn. 8 no.3:361-366 '65. (MIRA 18:10)

1. Moskovskiy institut tonkoy khimicheskoy tekhnologii imeni Lomonosova, kafedra khimii i tekhnologii redkikh i rasseyannykh elementov.

PLYUSHCHEV, V.Ye.; VARFOLOMEYEV, M.B.

Lanthanum perchlorates. Zhur. neorg. khim. 10 no.1, 199-208  
Ja '65. (MIRA 18:11)

1. Moskovskiy Institut tonkoy khimicheskoy tekhnologii imeni  
Lomonosova. Submitted Dec. 18, 1965.

PIYUSHCHEV, V.Ya.; VARFOLOMEYEV, M.B.

Yttrium perhenates. Zhur. neorg. khim. 10 no.1:109-114  
Ja '65. (MIRA 18:11)

1. Moskovskiy institut tonkoy khimicheskoy tekhnologii imeni  
Lomonosova. Submitted April 8, 1964.

SHAKHNO, I.V.; PLIUSHCHEV, V.Ye.; AVZHIYEVA, Ye.M.

The system  $\text{Na}_2\text{CrO}_4$  -  $\text{Rb}_2\text{CrO}_4$  -  $\text{H}_2\text{O}$  at 25 and 50°C. Zhur. neorg.  
khim. 10 no. 2: 552-555 F'65. (MIRA 18:11)

1. Moskovskiy institut tonkoy khimicheskoy tekhnologii imeni  
Lomonosova. Submitted April 10, 1964.

GRIZIK, A.A.; PLYUSHCHEV, V.Ye.

Rubidium polyhafnates. Zhur.neorg.khim. 10 no.12:2630-2638  
D '65. (MIRA 19:1)



PLYUSHCHEV, V.Ye.; LEPESHKOV, I.N.

Second All-Union Conference on Rare Alkaline Elements.  
Zhur.neorg.khim. 10 no.12:2845-2847 D '65.

(MIRA 19:1)

STEPIN, B.D.; FLYUSHCHEV, V.Ye.; FAKEYEV, A.A.

Anion halogenates of alkali metals and ammonium. Usp.khim.  
34 no.11:1881-1907 N '65.

(MIRA 19:1)

1. Vsesoyuznyy nauchno-issledovatel'skiy institut khimicheskikh  
reaktivov i osobo chistykh khimicheskikh veshchestv.

L 33244-65 EWT(m)/EWP(t)/EWP(b) IJP(c) JD/JG

ACCESSION NR: AP5005890

S/0020/65/160/003/0608/0611

AUTHOR: Komiesarova, L.N.; Varfolomeyev, M.B.; Ivanov, V.I.; Plyushchev, Y. Ye.TITLE: Synthesis and some properties of scandium perrhenates

SOURCE: AN SSSR. Doklady, v. 160, no. 3, 1965, 608-611

TOPIC TAGS: scandium perrhenate synthesis, anhydrous scandium perrhenate, scandium perrhenate monohydrate, scandium perrhenate trihydrate, scandium perrhenate stability, scandium perrhenate physical property

ABSTRACT: This is the first time that such compounds have been synthesized. Both the crystallohydrates and the anhydrous compound with the formula  $\text{Sc}(\text{ReO}_4)_3$  were prepared from  $\text{Sc}_2\text{O}_3$  and  $\text{NH}_4\text{ReO}_4$  with the addition of  $\text{HReO}_4$  (from metallic Re and nitric acid). Dissolution of the scandium oxide proceeded slowly with heat in the presence of hydrogen peroxide. An excess of  $\text{Sc}_2\text{O}_3$  afforded a rapid and complete reaction at an optimal pH of 4.5. Crystallization of the solution from which the  $\text{Sc}_2\text{O}_3$  excess had been filtered off occurred at 22-25 C. The process is described in detail. The products obtained,  $\text{Sc}(\text{ReO}_4)_3 \cdot 3\text{H}_2\text{O}$ ,  $\text{Sc}(\text{ReO}_4)_3 \cdot \text{H}_2\text{O}$  and  $\text{Sc}(\text{ReO}_4)_3$  were studied. The trihydrate, crystallizing in prisms at 25C, dissolves readily in water. Its crystals, viewed under X-ray, have a triclinial primitive cell. At 50 C the trihydrate loses 2

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ACCESSION NP: AP5005890

molecules of water and turns into a monohydrate, stable up to 140 C, after which it becomes anhydrous. The anhydrous form is stable up to 550 C. At higher temperatures, it slowly dissociates into  $\text{Sc}_2\text{O}_3$  and  $\text{R}_2\text{O}_7$ . Thermographic and thermogravimetric studies agreed satisfactorily with the data on isothermal dehydration. Results of diffraction studies are also reported. The anhydrous perhydrate is highly hygroscopic and rapidly changes into the monohydrate in air. Orig. art. has: 3 figures and 2 tables.

ASSOCIATION: Moskovskiy gosudarstvennyy universitet im. M. V. Lomonosova  
(Moscow State University)

SUBMITTED: 07Jul64

ENCL: 00

SUB CODE: IC

NO REF SOV: 002

OTHER: 000

Card 2/2

L 06580-67 EWT(m)/EWF(e)/EWP(t)/ETI IJP(c) AT/WH/JD/JQ  
ACC NR: AP6029822 SOURCE CODE: UR/0363/66/002/008/1460/1466

AUTHOR: Plyushchev, V. Ye.; Amosov, V. M.

ORG: Moscow Institute of Precision Chemical Technology im. M. V. Lomonosov  
(Moskovskiy institut tonkoy khimicheskoy tekhnologii)

TITLE: Synthesis and properties of the tungstates of erbium, thulium, ytterbium, and lutetium

SOURCE: AN SSSR. Izvestiya. Neorganicheskiye materialy, v. 2, no. 8, 1966, 1460-1466

TOPIC TAGS: lutetium, thulium, ytterbium, erbium, tungstate, phase composition, solid mechanical property

ABSTRACT: Kinetics of the interaction of W, Er, Tm, Yb, and Lu oxides was studied in 500°-1050°C range. The structure of the tungstates of the general formula  $M_2O_3 \cdot 3WO_3$  (where M is Er, Tm, Yb, or Lu) was determined by x ray technique and melting points, densities, and degrees of decomposition in various acidic and basic media (1 and 10 normal) were determined. The object of the work was to fill the gap in the pertinent literature. The various tungstate samples were prepared by grinding mixtures of  $M_2O_3$  with  $WO_3$  in 1:3 molar ratio followed by 15-480 minute fusion in 500°-1000°C range. The thermograms showed thermal effects at 540° and 778°C for  $Er_2O_3 + 3WO_3$ , 528° and 640°C for  $Tm_2O_3 + 3WO_3$ , 536° and 655°C for  $Yb_2O_3 + 3WO_3$ , and 508° and 655°C for  $Lu_2O_3 + 3WO_3$ . For Er,

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UDC: 546.65'786

ACC NR: AP6029822

Tm, Yb, and Lu tungstates, the respective melting temperatures ( $^{\circ}\text{C}$ ) and densities ( $\text{g}/\text{cm}^3$ ) were found to be:  $1500^{\circ}$  and 5.18,  $1520^{\circ}$  and 5.23,  $1540^{\circ}$  and 5.32, and  $1580^{\circ}$  and 5.34. At room temperature all tungstates were found to be stable when treated with diluted acids or bases but at  $80^{\circ}$ - $120^{\circ}\text{C}$  their decomposition was almost complete in 2 hrs. Orig. art. has: 4 figures and 2 tables.

SUB CODE: 07/ SUBM DATE: 29Jun65/ ORIG REF: 003/ OTH REF: 007

*ml*  
Card 2/2

ACC NR: AP6019045

(A)

SOURCE CODE: UR/0078/66/011/002/0294/0298

AUTHOR: Plyushchev, V. Ye.; Varfolomeyev, M. B.ORG: Moscow Institute of Fine Chemical Technology im. M. V. Lomonosov (Moskovsky institut tonkoy khimicheskoy tekhnologii)TITLE: Perrhenates of neodymium and samarium

SOURCE: Zhurnal neorganicheskoy khimii, v. 11, no. 2, 1966, 294-298

TOPIC TAGS: rhenium compound, neodymium compound, samarium compound, inorganic synthesis, refractive index, melting point

ABSTRACT: The synthesis of neodymium and samarium perrhenates was made with an excess of  $\text{Me}_2\text{O}_3$  ( $\text{Me} = \text{Nb}$  or  $\text{Sm}$ ) by dissolving oxide ( $\sim 2.5$  g  $\text{Nd}_2\text{O}_3$  or  $\text{Sm}_2\text{O}_3$ ) in  $\text{HReO}_4$  ( $\sim 70$  ml at  $\sim 240$  g/l concentration (heated in a water bath). The reaction was controlled by methyl red (pH of transition was 6.2) and the  $\text{H}_2\text{O}_2$  was added to accelerate the reaction. The solutions obtained were filtered out for removal of excess  $\text{Me}_2\text{O}_3$  and the compounds were crystallized by steaming the solutions in a water bath at  $75-80^\circ\text{C}$ . The  $\text{Nd}(\text{ReO}_4)_3 \cdot \text{H}_2\text{O}$  and  $\text{Sm}(\text{ReO}_4)_3 \cdot \text{H}_2\text{O}$  were formed under these conditions when the solutions were left to evaporate completely. The crystals, consisting of  $\text{Nd}(\text{ReO}_4)_3 \cdot 4\text{H}_2\text{O}$  and  $\text{Sm}(\text{ReO}_4)_3 \cdot 4\text{H}_2\text{O}$ , were produced when the solutions were evaporated to almost saturated conditions and cooled to room temperature. The crystals of  $\text{Me}(\text{ReO}_4)_3 \cdot \text{H}_2\text{O}$  and

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UDC: 546.719.7'657+546.719.7'659

ACC NR: AP6019045

$\text{Me}(\text{ReO}_4)_3 \cdot 4\text{H}_2\text{O}$  were nonhygroscopic, soluble in water and alcohol, and partly weathered during prolonged storage in air. The neodymium and samarium perhenates had an orthorhombic habit and the following properties:

	Density g/cc	Refractive $\gamma$	Indexes $\alpha$
Nd( $\text{ReO}_4$ ) <sub>3</sub> ·4H <sub>2</sub> O	4.395	1.675	1.668
Sm( $\text{ReO}_4$ ) <sub>3</sub> ·4H <sub>2</sub> O	4.487	1.684	1.675
Nd( $\text{ReO}_4$ ) <sub>3</sub> ·H <sub>2</sub> O	5.285	1.733	1.720
Sm( $\text{ReO}_4$ ) <sub>3</sub> ·H <sub>2</sub> O	5.331	1.733	1.723

Dehydration of  $\text{Me}(\text{ReO}_4)_3 \cdot 4\text{H}_2\text{O}$  at 50C resulted in their transformation into  $\text{Me}(\text{ReO}_4)_3 \cdot \text{H}_2\text{O}$ . The latter was converted at 140-160C into anhydrous salt. The anhydrous neodymium and samarium perhenates were strongly hygroscopic, easily soluble in water and alcohol, and had refractive indexes higher than those of the standard set of immersion liquids. The anhydrous perhenates were stable during heating to temperatures below 650C. They decomposed at temperatures > 650C with liberation of  $\text{Re}_2\text{O}_7$ :  $2\text{Me}(\text{ReO}_4)_3 \rightarrow \text{Me}_2\text{O}_3 + 3\text{Re}_2\text{O}_7$ . The decomposition was very slow, and slightly decomposed anhydrous neodymium and samarium perhenates melted at 952 and 905C, respectively. Orig. art. has: 5 fig. and 6 tables.

SUB CODE: 07/ SUBM DATE: 09Nov64/ ORIG REF: 003/ OTH REF: 001

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L 42883-66 EWP(e)/EWT(m)/I/EWP(t)/ETI/EWP(k) JLP(s) JD/JG  
ACC NR: AP6022892 SOURCE CODE: UR/0078/66/011/004/0732/0737 318

AUTHOR: Plyushchev, V. Ye.; Amosov, V. M.

ORG: none

TITLE: Synthesis and properties of neutral yttrium tungstate

SOURCE: Zhurnal neorganicheskoy khimii, v. 11, no. 4, 1966, 732-737

TOPIC TAGS: tungstate; yttrium compound

ABSTRACT: Yttrium tungstate,  $Y_2O_3 \cdot 3WO_3$ , was synthesized by sintering the powdered oxides at 600-1000°C. The extent of the reaction (amount of reacted  $WO_3$ ) was checked by chemical quantitative phase analysis. The reaction between  $Y_2O_3$  and  $WO_3$  was also studied by differential-thermal (Kurnakov pyrometer) and x-ray phase analyses. The rate of this reaction was found to be determined by the diffusion of the initial oxides through the layer of the reaction products. The reaction begins only after the temperature of polymorphic transformation of  $WO_3$  is reached at 750°C, and practically reaches completion at 900°C. Yttrium tungstate is an anisotropic, finely crystalline, colorless substance insoluble in water, alcohol, and acetone. Its density is 4.407 g/cm<sup>3</sup>, and its melting point, 1470 ± 20°C. It does not decompose at least up to the melting point, and does not show any transformations. The interplanar distances and x-ray line intensities of yttrium tungstate are given. The action of acids and

UDC: 546.786'643-31

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ACC NR: AP6022892

alkalis on this compound was determined. Orig. art. has: 5 figures and 2 tables.

SUB CODE: 07/ SUBM DATE: 30Jan65/ ORIG REF: 003/ OTH REF: 008

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L 36959-66 EWT(m)/EWP(1)/EWP(t)/ETI IJP(c) JD/JG/RM  
 ACC NR: AP6014891 SOURCE CODE: UR/0076/65/039/012/2924/2926 38  
 AUTHOR: Shklover, L. P.; Plyushchev, V. Ye. B  
 ORG: All-Union Scientific Research Institute for Chemical Reagents and  
 Very Pure Chemical Substances (Vsesoyuznyy nauchno-issledovatel'skiy  
 institut khimicheskikh reaktivov i osobo chistyykh khimicheskikh  
 veshchestv)  
 TITLE: The strength of the metal bond in the phthalocyanines of the  
 rare earth elements 21  
 SOURCE: Zhurnal fizicheskoy khimii, v. 39, no. 12, 1965, 2924-2926  
 TOPIC TAGS: rare earth element, spectrophotometric analysis  
 ABSTRACT: Metal phthalocyanines are usually characterized by a single  
 intense (long wave) absorption band in the visible region of the  
 spectrum, the position of which in a given solvent depends on the nature  
 of the central metal. Spectroscopic investigations were made of the  
 position of this maximum absorption band for 17 different rare earth  
 elements. The experimental results are shown in a table. The data in  
 the table show that the direction of the displacement of the position of  
 the maximum absorption band of the phthalocyanines of the metals in the  
 UDC: 541.20  
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L 20612-66 EWT(m)/ENP(j)/T RM

ACC NR: AP6010752

SOURCE CODE: UR/0076/66/040/003/0741/0743

AUTHOR: Nasirdinov, S. B.; Shugam, Ye. A.; Berger, L. I.; Plyushchev, V. Ye.; Shklover, L. P. 47  
B

ORG: All-Union Scientific Research Institute of Chemical Reagents and High Purity Chemicals (Vsesoyuznyy nauchno-issledovatel'skiy institut khimicheskikh reaktivov i osobo chistykh khimicheskikh veshchestv)

TITLE: Electrical conductivity of phthalocyanines of certain metals

SOURCE: Zhurnal fizicheskoy khimii, v. 40, no. 3, 1966, 741-743

TOPIC TAGS: organic semiconductor, phthalocyanine, electrical conductivity

ABSTRACT: The effect of the metal atom on the thermal activation energy for conduction of certain metal-containing phthalocyanines has been studied and the activation energy was correlated with the position of the peak in the long wavelength (320 to 1100 mμ) region of the absorption spectrum. To this end, the temperature dependence of conductivity was measured and absorption spectra were recorded for phthalocyanines of transition metals of groups IV (titanium, zirconium, and hafnium) and VIII (nickel, palladium, and platinum) of the periodic table. Chloro derivatives of the phthalocyanines were used in all cases except that of nickel. Electrical measurements were carried out for pellet samples in vacuum ( $10^{-3}$  mm Hg) at 25 to 230C. It was found that the temperature dependence of conductivity obeyed an exponential law in all

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ACC NR: AP6010752

cases. Electrical conductivities varied from  $5 \times 10^{-13}$  to  $2 \times 10^{-10}$  mho/cm and activation energies from 1.35 to 1.60 ev. The activation energy and the energy required for transition of the chlorophthalocyanines changed symbatically and rose with increasing atomic number of the metal. It is suggested that this rise is associated with an increase in the donor-acceptor interaction between the metal atom and nitrogen atoms in the phthalocyanines. The energy of electrons in the conducting state was lower than the energy of the lowest excited state. [SM]

SUB CODE: 20, 11/ SUBM DATE: 23Jul65/ ORIG REF: 006/ OTH REF: 007/ ATD PRESS:

4124

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BK

L 13640-66 EWT(m)/ETC(F)/EPF(n)-2/EWG(m)/EWP(t)/EWP(z)/EWP(b) IJP(c)  
 ACC NR: AP6000765 DS/JD/WW/HW/JG/RM SOURCE CODE: UR/0078/65/010/012/2845/2847  
 AUTHOR: Plyushchev, V. Ye.; Lepeshkov, I. N.  
 ORG: none  
 TITLE: Second ~~all~~-union conference on the rare alkali metals  
 SOURCE: Zhurnal neorganicheskoy khimii, v. 10, no. 12, 1965, 2845-2847  
 TOPIC TAGS: metallurgic conference, chemical conference, alkali metal, cesium, lithium, rubidium, metal compound, metal extraction, metal purification, ion exchange, inorganic synthesis, chemical bonding, polyhalide salt, phase diagram, solubility, aqueous solution  
 ABSTRACT: The Second All-Union Conference on Rare Alkali Metals was held 13—16 October 1964 in Novosibirsk and was jointly sponsored by the Siberian Department and Department of Physical Chemistry and Technology of Inorganic Materials of the AS USSR, the USSR State Committee on Coordination of Scientific Research, and the Novosibirsk section of the All-Union Chemical Society im. D. I. Mendeleev. More than 50 scientific papers were presented which dealt with general subjects of the synthesis and properties of the rare alkali metal compounds, methods of determination, extraction, separation, and purification of the rare alkali metals, chemical

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ACC NR: AP6000765

interactions in aqueous solutions and melts of their compounds, and applications of these metals and their compounds. There was a significant number of papers on the synthesis and properties of the compounds. The following topics were noted in this category: new data on zirconates and hafnates of alkali metals (Plyushchev, V. Ye., and A. A. Grizik); fluorozirconates and fluorohafnates of Rb and Cs (Tananayev, I. V., and L. S. Guzeyeva); reactions of hydroxides and carbonates of the rare alkali metals with hydrogen peroxide (Dobrynina, T. A., B. S. Dzyatkevich, N. A. Akhapkina, and A. I. Chernysheva); cesium superoxide and ozonide (Vol'nov, I. I., and V. V. Matveyev); thermal stability of Rb and Cs compounds (Breusov, O. N., N. I. Kashina, G. V. Revzin, N. A. Druz', A. O. Lesovaya, P. D. Komissarova, and R. M. Shklovskaya); lithium aluminumhydride and galliumhydride (Arkhipov, S. M., and V. I. Mikheyeva); lithium and boron complexes (Arkhipov, S. M., G. Ye. Revzin, and P. D. Komissarova); ion-exchange properties of ferrocyanides and application in the extraction of the rare alkali metals (Kozlov, A. S., and Yunden Mizhidiyn); ion-exchange property of granulated nickel and alkali metal ferrocyanides (Vol'khin, V. V., S. A. Kolesova, and A. F. Kalashnikova); amalgam method of preparing Rb and Cs hydroxides (Sklyarenko, S. I., A. P. Rysev, and I. V. Smirnov); and Li reduction on the liquid gallium cathode (Ponomarev, V. D.).

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Y. V., V. P. Porubayev, and A. I. Zazubin). Three papers were given on Rb and Cs determination by different methods. Nine papers dealt with selective extraction of the rare alkali metals from raw materials, six of them with Li, one with Rb, and two with unspecified alkali metals. The following papers were noted in the series on separation and purification: separation of the rare alkali metals by continuous counter-current ion-exchange (Gorshkov, V. I., G. M. Panchenkov, G. M. Gulyayeva, S. N. Dmitriyev, N. N. Savenkova, and G. M. Medvedev); separation of Li from certain alkali and alkali earth metals by ion-exchange (Suvorovskaya, N. A., V. V. Shikhova, and I. A. Shmarinova); purification of Rb and Cs compounds by crystallization (Vulikh, A. I., A. O. Lesovaya, V. A. Kaz'minskaya, L. P. Zherdiyenko, S. M. Arkhipov, and R. M. Shklovskaya); and preparation of high-purity Cs and its compounds (Gulyayeva, G. M.). The papers on melts and aqueous solutions of salts of the rare alkali metals attracted special attention. G. V. Samsonov reported on the nature of the chemical bond and phase structure of the rare alkali metal compounds with non-metallic elements and indicated directions to follow in the synthesis of these compounds with given properties. Two papers were noted on the phase diagram of fused salt systems  $\text{MeCl}_3\text{—RbCl}$  and  $\text{MeCl}_3\text{—CsCl}$ , where Me = Ga or In (Arbekov, V. N., V. A. Sryvtsev, and Ye. S. Petrov), and  $\text{MeCl—ScCl}_3$ , where

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Me = Li, Rb, or Cs (Fedorov, N. Ya., and Ye. S. Petrov). The aqueous salt systems were treated in several papers, one which was on solubility at 25C in the Cs compound-K compound-H<sub>2</sub>O system (Kirgintsev, A. N., and L. N. Trushnikova). Three papers dealt with polyhalide systems as a means of purification of Rb salts (Kuznetsova, G. P., V. I. Safonova, and B. D. Stepin; Fakeyev, A. A., V. N. Kulyukin, and B. D. Stepin) or Rb and Cs salts (Stepin, B. D., A. V. Babkov, and T. M. Sas). Two of several papers were noted in the category of applications: vitreous lithium silicates (Dubrovo, S. K.) and the effect of Rb and Cs oxides on the properties of vitreous and crystallized silicates (Alekseyeva, Z. D.). The Third All-Union Conference will be held in 1968. [ATD PRESS: 4169-F]

SUB CODE: 11, 07 / SUBM DATE: none

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GRIZIK, A.A.; PLYUSHCHEV, V.Ye.

Rubidium trizirconate. Zhur. neorg. khim. 10 no.9:1993-2000 S '65.  
(MIRA 18:10)

PLYUSHCHEV, V.Ye.; AMOSOV, V.M.

Synthesis and properties of neutral lanthanum, cerium, praseodymium,  
and neodymium tungstates. Izv. AN SSSR. Neorg. mat. 1 no.7:1155-1161  
Jl '65. (MIRA 18:9)

1. Moskovskiy institut tekhnicheskoy khimicheskoy tekhnologii imeni M.V.  
Lomonosova.

L 4457-66 EWT(m)/EWP(t)/EWP(b) IJP(c) JD/JG

ACCESSION NR: AP5018718

UR/0070/65/010/004/0509/0514  
548.736:535.342

AUTHORS: Ivanov, V. I.; Varfolomeyev, M. B.; Petrov, K. I.;  
Pervykh, V. G.; Plyushchev, V. Ye.

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55  
B

TITLE: X-ray diffraction and infrared spectroscopic study of  
tetrahydrates of perrhenate of rare earth elements and yttrium

SOURCE: Kristallografiya, v. 10, no. 4, 1965, 509-514

TOPIC TAGS: x-ray diffraction analysis, IR spectroscopy, crystal  
lattice structure, crystal symmetry, crystal unit cell, rare earth  
element

ABSTRACT: The authors investigated crystals of tetrahydrates of  
perrhenate of lanthanum, lanthanoids, and yttrium, the production and  
chemical analysis of which were described in an earlier paper (Dokl.  
AN SSSR v. 158, 664, 1964). A schematic study of the single crystals  
in x-ray cameras and with a diffractometer has shown that these sub-  
stances crystallize in three different structural types. The syngony,

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